REMARKS

Claims 1-27 are presently pending. The Examiner has indicated that claims 11 and 12 are allowed and that claims 7, 8, 20 and 24-26 would be allowable if rewritten to include all of the limitations of the claims from which they depend.

Applicants respectfully request reconsideration of the application in view of the remarks appearing below, which Applicants believe demonstrate that the application is in condition for allowance.

Rejection under 35 U.S.C. § 103

The Examiner has rejected claims 1-6, 9, 10, 13-19, 21-23 and 27 under 35 U.S.C. § 103 as being obvious in view of the Briggs patent and the Moura Bordado et al. publication, both of which are addressed in detail in the prior Amendment filed on November 24, 2004. More particularly, the Examiner asserts that Briggs discloses all of the limitations of these claims except for: (1) the testing of cork stoppers for the presence of TCA; (2) an apparatus for accomplishing the same and (3) particular features of the apparatus. The Examiner then states that Moura Bordado et al. discloses the testing of cork stoppers for the presence of TCA and asserts that it would have been obvious to a person having ordinary skill in the art at the time of the invention, in view of the Moura Bordado et al. publication, to test cork stoppers and provide a cork stopper testing apparatus or method as contemplated by the rejected claims. Applicants respectfully disagree.

The Briggs/Moura Bordado et al. Combination Lacks a Sensor Operatively Configured to Detect an Analyte that Causes Cork Taint in Wine

First, Applicants desire it to be clear that Moura Bordado et al. do not disclose or suggest the testing of cork stoppers for the presence of TCA by sniff testing the stoppers themselves. Rather, Moura Bordado et al. explicitly state at page 11, lines 23-25 that cork samples were analyzed by immersing the stoppers individually in 100 mL of white wine for a period of 24 hours, after which the individual wine samples were subject to an olfactory assessment (hereinafter "sniff test") by a minimum of three human assessors. In this sniff test, olfactory

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characteristics of the wine samples were compared against one another. It is noted that multiple human assessors are required to account for inaccuracies in, and subjectivity of, human sensory assessment. The slow and tedious human-based method of testing cork stoppers disclosed by Moura Bordado et al. is vastly different from the present invention that can efficiently, objectively and quickly test every cork stopper in virtually any number of batches without the need to deal with the subjectivity and variation of sensory perception that accompanies human assessment.

Applicants point out this distinction because any combination of the Briggs and Moura Bordado et al. references fails to disclose a sensor operatively configured to detect the presence of an analyte responsible for cork taint in wine as required by all of the rejected claims. Applicants respectfully assert that such sensors were not so well-known prior to the present invention that the Examiner can simply assert that such sensors are indeed well-known without providing evidence. Consequently, Applicants believe that the Examiner must provide evidence of such a sensor in order to present a *prima facie* case of obviousness.

Because any combination of the Briggs and Moura Bordado et al. references lacks the required sensor, the rejected claims cannot be rendered obvious by this combination.

As discussed below in detail, the Moura Bordado et al. method of reducing the level of TCA in cork stoppers is essentially yet another solution for dealing with cork taint issues in the absence of any viable technology for testing each and every cork stopper that is intended for use in bottling wine. Generally, the Moura Bordado et al. method provides a means for treating cork stoppers en mass, regardless of whether or not the stoppers actually contain TCA. In general, the primary conventional method of providing quality control for cork stoppers relative to cork taint is to take a statistical sample from each batch of stoppers or cork sheets used to make stoppers. If the sample is tainted, the entire batch is identified as being tainted, regardless of whether or not in fact the batch contains untainted stoppers or sheets. The Moura Bordado et al. method simply provides a way of treating the batches identified as being tainted. The conventional statistical sampling method, even in combination with the Moura Bordado et al. removal method, does not address whatsoever the underlying problem that, except for the present invention, no

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one has yet devised an efficient and fast method of testing every single cork stopper desired to be used.

The Invention of Rejected Claims 1-6, 9, 10, 13-19, 21-23 and 27 Satisfies a Long Felt but Unsolved Need to Test Every Cork Stopper Intended for Use

As the Examiner knows, it is the policy of the U.S. Patent and Trademark Office to follow the obviousness analysis laid out in Graham v. John Deere Co., 383 U.S. 1, 148 USPQ 459 (1966), including the evaluation of secondary considerations of nonobviousness, which include long-felt but unresolved needs. MPEP § 2141. Even when it appears that the prior art suggests that a claimed invention may be obvious, one or more secondary considerations of nonobviousness may support a conclusion that the invention is in fact not obvious.

In the present case, the invention recited in the rejected claims indeed satisfies a long felt but unsolved need in the wine industry to test each and every cork stopper that is intended to be used to bottle wine. In particular, e.g., claim 1 as previously amended includes the limitations of moving, in seriatim, first and second cork stoppers to a first position and also moving first and second sensors to a second position wherein the sensors are used to determine whether or not, respectively, the first and second cork stoppers contain an analyte that causes cork taint in wine. As discussed below, it is the cycling of the first and second sensors that allows the present invention to test each and every cork stopper that is desired to be used to bottle wine. In fact, claim 2, which was also previously amended, particularly captures the concept of testing each and every cork stopper in a plurality of batches of stoppers, a feat that has not yet been achieved in the wine industry despite the fact that automated sensing technologies has existed in other industries for many years.

As described below in detail, despite all of the sophisticated testing techniques utilized for testing cork and cork stoppers for the presence of TCA, to the best of Applicants' knowledge, all still rely on statistical sampling, which by its very nature permits at least some untainted cork or cork stoppers to be identified as unsuitable for use and, therefore, must be wasted. The testing of all cork stoppers destined for use would combat such waste. Applicants assert that this

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secondary consideration of nonobviousness is so strong that the U.S. Patent and Trademark Office should allow Applicants to protect their invention, which is directed specifically to the wine industry, despite the fact that other industries may implement systems and methods that utilize a similar type of equipment, but for much different uses.

As mentioned above and as discussed in the prior Amendment filed on November 11, 2004, the best practices conventionally used to provide the wine industry with quality control as it relates to inhibiting the occurrence of cork taint in wine involve the random statistical sampling of batches in which only a portion of the cork stoppers or bulk cork is tested. Typically, this involves random removal of a few stoppers, or "test stoppers," from each batch. The test stoppers are then relatively painstakingly tested using conventional sniff testing or involved chemical analyses, such as gas chromatography, mass spectroscopy and solid phase microcxtraction (SPME). Conventional sniff testing is often performed by highly trained human sniffers.

In order to illustrate the recent history of quality control in connection with cork taint of wine, Applicants have attached hereto as Exhibits A-E a number of publications widely available, e.g., on the World Wide Web. Exhibit A is a printout of a Web page of Scott Laboratories, a manufacturer of equipment, supplier of cork and provider of an array of services to the wine making industry, among other industries. Exhibit A indicates that Scott Laboratories was the first cork supplier to institute sniff testing for TCA. Scott Laboratories first instituted TCA sniff testing in 1984.

Exhibit B is an article from the June 1996 issue of the publication Wine Business Monthly that described the state-of-the-art quality-control testing implemented for cork stoppers in the wine industry at the time. As the article explains, such testing included human sniff testing of only random samples, i.e., small numbers, of cork stoppers and human sniff and taste testing of wine subjected to sampled stoppers. These slow, incomplete (only a small fraction actually tested), subjective, destructive (the sampled cork cannot be reused) and painstaking testing techniques are far removed from the claimed invention, which, by virtue of its claimed multiple sensor arrangement and multiple sensor method is fast, complete (every cork stopper

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may be tested), objective, nondestructive and straightforward. In other words, the claimed invention is a vast and much needed improvement over the testing techniques implemented in 1996. Despite the passage of 12 years, i.e., from 1984 to 1996, since sniff testing was implemented, no one implemented a system and method able to test each and every cork stopper destined for use.

Next, Exhibit C is a printout of Scott Laboratories "A Commitment to Quality" brochure available at www.scottlaboratories.com/info-center/pdf/CorkQualityCommitment.pdf that is accessible from the Web page at www.scottlaboratories.com/info-center/faq-packaging.asp, which is included as Exhibit D. As seen from Exhibit D, access to the "A Commitment to Quality" brochure is accessible from the question "What does Scott Labs do to prevent TCA (cork taint)?" on the Web page.

As seen in Exhibit C, the "A Commitment to Quality" brochure, which was downloaded and printed on April 7, 2005, describes the (presumably) state-of-the-art practices that Scott Laboratories presently implements as its quality control measures relative to TCA testing of cork. The brochure appears to indicate that SPME, a technique apparently invented in the early 1990s (see Exhibit E), represents the state-of-the-art in TCA testing in 2005. Applicants particularly note that the brochure of Exhibit C indicates that, despite the use of the sophisticated SPME testing method, testing still involves random statistical sampling. Consequently, even in today's state-of-the-art TCA testing, there remains no practical way to test each and every cork stopper effectively. In sharp contrast, the claimed invention, which, again, utilizes a multi-sensor arrangement that is unique to the wine industry, allows for the testing of all cork stoppers slated for use. This is a feat that the wine industry in all of its years of existence has not been able to achieve.

Applicants particularly note that in the 21 years (1984 to 2005) since Scott Laboratories was the first to implement sniff testing for TCA, it appears that no one, except for Applicants, has designed any system and method that can quickly, completely, objectively, nondestructively and easily test all cork stoppers that are intended to be used to bottle wine. This is so, despite the

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fact that references such as the Briggs patent exist and are available to appropriately skilled artisans.

In view of the foregoing, as of 2005 there remains a long felt, yet unsolved, need for a system and method for testing cork stoppers for the presence of an analyte that causes taint in wine in each and every cork stopper intended to be used to cork bottles of wine so that uncontaminated cork stoppers present in batches designated as tainted using conventional random sampling can be used. The system and method claimed in the rejected claims satisfies this long felt need.

For at least the foregoing reasons, Applicants respectfully request that the Examiner withdraw the present obviousness-type rejection of claims 1-6, 9, 10, 13-19, 21-23 and 27.

CONCLUSION

In view of the foregoing, Applicants submit that claims 1-27, as previously amended, are in condition for allowance. Therefore, prompt issuance of a Notice of Allowance is respectfully solicited. If any issues remain, the Examiner is encouraged to call the undersigned attorney at the number listed below.

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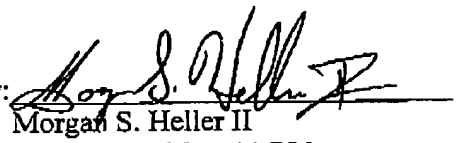
Respectfully submitted,

MICHAEL S. HEAD ET AL.

By: 
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Attachments
Exhibits A-E

BTV.434784.1

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Thursday, April 7, 2005

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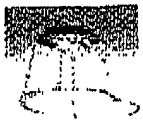
PACKAGING – CORKS


Corks


Synthetic Corks


Champagne Corks


Wire Hoods


Aluminum Screwcaps

Scott Laboratories has been supplying still wine corks to the North American market for almost three decades. North American firsts include:

- First cork supplier to incorporate laboratory testing of corks (1977)
- First supplier to package corks under SO_2 in hermetically-sealed plastic bags (1978)
- First cork supplier to sample every bale for quality upon receipt from Portugal (1980)
- First cork supplier to use a humidity controlled warehouse (1981)
- First cork supplier to institute "sniff testing" for TCA (1984)
- Founding member of the Cork Quality Council (1992)
- First supplier to institute a SPME quality control program (1999)

Cork quality must start in Portugal. All corks go through rigorous checks in our ISO certified warehouse and laboratory in Portugal prior to shipment. All lots are prescreened using our SPME units in Portugal.

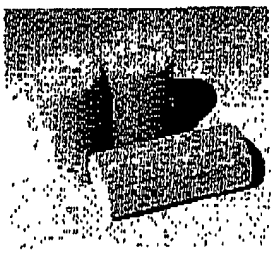
The corks are later subjected to further rigorous testing at our ISO certified facility in California. All testing meets or exceeds Cork Quality Council standards. All lots are subjected to SPME testing for TCA by ETS. Storage before processing is done in our humidity controlled warehouse which is subject to ozone nightly.

Every effort is made to provide you with the best possible corks. Only after they pass our standards are they made available to you. Please see our Commitment to Quality.

Minimum order is 1,000 corks.

SPECIFICATIONS

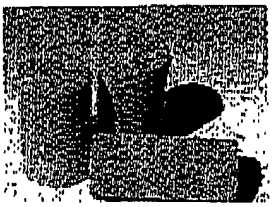
Sterisun Corks



These natural corks have received a light hydrogen peroxide and water wash followed by neutralizing rinses. Corks are then dried to a moisture level between 5 and 8%. They are, quite simply, the cork standard.

- Lengths: 54 mm, 49 mm, 45 mm, 38 mm
- Diameter: 24 mm
- Qualities: USS, US+,US, UFS, UF, UFB, UFB1, UFBB, UFB4

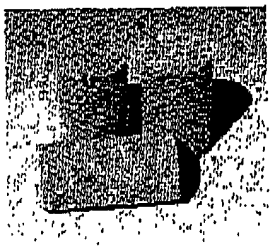
Natural Corks



These natural corks have received a light potassium metabisulfite and water wash. Corks are then dried to a moisture level between 5 and 8%.

- Lengths: 54 mm, 49 mm, 45 mm
- Diameter: 24 mm
- Qualities: USS, US+,US, UFS, UF

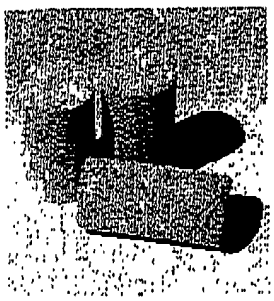
One + One Corks



Combining natural cork ends with an agglomerated middle makes this cork an economic alternative.

- Lengths: 45 mm, 38 mm
- Diameter: 23.5 mm
- Qualities: US (A), UF (B), UFB1 (C)

Colmated Corks



Manufactured by using a combination of food grade adhesive and cork dust to fill imperfections. These natural corks provide an attractive, low cost alternative.

- Length: 45 mm
- Diameter: 24 mm
- Qualities: UFB1, UFBB, UFB4, UFB5

Bar and Bulb Top Corks

These natural cork shafts with plastic top are available upon request.

*All corks stored in a moisture-controlled area with microbial growth held in check using ozone prior to processing.

*All corks are dedusted, branded, processed and packed under SO_2 in our modern and efficient cork processing facility.

*Cork Printing - A printing die is made from winery-supplied artwork. This die is inked and transferred onto the cork for a custom design.

Cork Suppliers Strive For Inproved Quality

This article originally appeared in the June 1996 issue of the Wine Business Monthly.

CORK SUPPLIERS STRIVE FOR IMPROVED QUALITY
Extensive Laboratory Testing Produces Better Product

By Teri Shore
Staff Writer

US. cork suppliers are spending more time and resources in labor-intensive sampling and evaluation to prevent cork taint and assure vintners of good cork quality.

Cork Supply USA in Benicia, the largest supplier of corks in the United States, recently expanded its laboratory, testing methods and staff to assure increased levels of cork quality.

Cork Supply USA now employs three laboratory clinicians who test every lot that comes into the warehouse from producers in Portugal.

"We've doubled the size of our laboratory and added new equipment," said Justin Davis, sales manager. "The additions give us the capability to do extensive coating research and experimentation, and more accurate microbial testing."

Juvenal Direct in Napa also uses extensive laboratory testing to assure high levels of cork quality. This Portuguese-owned company opened a facility in the United States last fall.

Winemakers have been dropping by Cork Supply USA lately to view the new facility, including a team from Beringer Winery that came by unannounced to inspect the laboratory_on a day when Cork Supply happened to be printing an order of corks for the winery.

The laboratory improvements include:

- A functional testing room with an automatic vacuum-head corker, an automatic cork extractor, and pressurized leak tester.
- A new clean room with fume hood, autoclave, incubator and oven.

New Extraction Testing

The extractor is a pneumatic device that extracts the cork and indicates the force required to do so. The extraction may be performed on corked bottles or on the plastic fixtures from the pressurized leak tester_a carousel apparatus used to evaluate the sealability of treated corks.

The extractor helps determine whether the amount of treatment applied was sufficient to allow removal of the cork without undue or excessive force. It is also used to establish treatment levels for applications using new glass and different bottle profiles. Problem bottles can be analyzed for extraction force and measured for conformity to specifications.

Carousel Evaluation

The carousel consists of a series of ports where corked fixtures can be attached and pressure applied to see if corks will leak. The fixtures are made of clear plastic with an internal diameter of 19 mm. The fixtures are placed in a custom-designed bottle which allows the cork to be inserted using the automatic vacuumed corker. The fixtures are corked and

allowed to stabilize for a minimum of 30 minutes, at which time wine is introduced through the other end, then attached to the carousel port. Nitrogen gas is used to supply the desired pressure. The pressure is brought up to 15 psi and the fixture remains under pressure for one hour. After the pressure is released the corks are removed and any leakers identified. The cork is extracted and evaluated to determine the cause of leakage.

The bulk of the laboratory's main room is taken up with grading and sensory evaluation sections, staffed by two technicians and managed by Devin Callaway, quality assurance manager.

Five Steps To Cork Quality

Callaway and her team use five standard inspection procedures to ensure cork quality: sensory evaluation, quality evaluation, residual oxidants testing, moisture measurement and physical characteristics.

Some of these steps may have already been taken at Cork Supply's sister company in

Portugal, Global Cork. Cork Supply Portugal supplies about 20 percent of Cork Supply USA's cork, with the rest purchased from independent producers.

The Global Cork lab evaluates the quality of the raw corks, both for Cork Supply and other producers who seek the testing expertise of the state-of-the-art laboratory in Portugal, explained Callaway.

Each year Global Cork tests more than 1 million corks per year out of 250 million corks the cork supply group purchases annually. More than 90 percent of all cork tested is rejected as unsuitable on first testing, she said.

Corks that pass the quality standards at Global Cork are tested again at Cork Supply USA headquarters to maintain quality continuity during

processing and treatment, which includes dedusting, moisturizing, printing, coating and packaging for delivery.

Each lot of corks purchased by a winery comes with three reports: a pre-shipment inspection report from Portugal, an incoming inspection report by Cork Supply USA and a final inspection report. The reports provide quantitative information on visual, sensory, and quality measurements.

Sensory Evaluation

Sensory evaluation begins when corks arrive in bags of 10,000, each shipped in large bales on container ships from Portugal.

A sniff test is performed on a random sampling of corks based on the size of the shipment. The bale sample is sniffed to ascertain the presence of off aromas and general sensory character. A gross quality evaluation is also made to identify potential problems.

Soak tests are then performed on the cork sampling. Cork Supply USA has expanded its soak test to include sensory evaluations of eight-to-10 individual corks from a bale.

A tasting team of seven people evaluates the samples in a procedure used more often at the winery. The individual soaks are in addition to the standard practice of group soaks, where three corks per bale sampled are placed in a 250 ml schott bottle and immersed in white wine.

Cork Supply recently switched from Carlo Rossi Chablis to Franzia Mountain Chablis because the latter is more sensitive to picking up off-characteristics, said Callaway.

After soaking for 24 hours, a portion of the wine is poured into a wine glass and sniffed to determine acceptability.

The results from each bottle are graded as follows:

1. clean, positive or neutral
2. slightly off character
3. strong off character
4. TCA

Any bales found to have tainted corks are placed on hold and are resampled. Fifty individual soak tests are performed. One in 50 corks with TCA is acceptable, but if two in 50 are found, the bale is rejected.

Residual Oxidants

The residual oxidants test detects the presence of oxidants on the cork surface.

Oxidants left on the cork after initial washing can cause oxidation in wines.

At Cork Supply USA, a solution of potassium iodide and acetic acid with a starch indicator is used to detect oxidants. If residual oxidants are present, the solution will turn a distinct violet color.

Cork Supply's Alpha-type corks are washed with a mild hydrogen peroxide solution and must remain unchanged in color to pass the test. Chlorine-washed corks are evaluated on a reference only basis and faint color changes are not uncommon.

Natura-type corks washed with potassium metabisulfite are also tested.

Moisture

Moisture samples for incoming lots are pulled from the bales' sample bags at a rate of 5 per bag, with a minimum of 15 corks and a maximum of 50 corks. A DC-2011 moisture meter is used to test the moisture levels by placing sensory needles into the side of each cork at one location. The median point is identified and documented. This evaluation is a reference only rather than a pass/fail test because moisture is monitored and adjusted before processing is complete.

Moisture is evaluated by production staff before the cork processing begins in the dedust area. It is checked again before and after treatment.

Quality Evaluation

After sensory, residual oxidants and moisture samples are pulled from the sample bags, the remaining corks are combined for grading.

They are segregated into letter categories, A through D. Points are assigned to each letter category and the point total for the lot is calculated and expressed per 100 corks.

Cork Supply uses intensive hand-selection for wineries that require a specific grading uniformity.

Physical characteristics of the corks are also checked at this time to make sure they are the correct length, diameter and shape.

Post-Treatment Tests; Examined for Capillary Activity

Corks are evaluated after treatment for capillary activity. They are tested at a rate of about four corks per 100,000 corks.

Cork Suppliers Strive For Inproved Quality

They are pulled randomly and placed on end in red wine at a height of about 5 ml.

The corks remain in the wine for 24 hours, after which they are inspected for wine travel up the cork.

After corks are bagged for shipment in plastic bags gassed with sulfur dioxide, they are tested once more 24 hours after packaging.

The test for microbiologic activity is performed once a day on larger orders which will be shipped after evaluation. Gas levels are also evaluated twice a day.

SCOTT LABORATORIES

A COMMITMENT TO QUALITY

Scott Laboratories, Inc. has been importing and processing corks longer than any current North American supplier.

- In the spring of 1978 Scott introduced the first corks packed in California under sulfur dioxide [with the trade name "Steriseal" ®].
- In 1982 Scott was the first American supplier to react to the Swiss study revealing TCA as a primary culprit in cork taint. Rather than denying TCA's existence, Scott actively distributed vials to wineries to increase industry awareness. Soon thereafter, Scott initiated the American cork industry's first sensory screening program for incoming corks.
- Always pushing for strict quality standards, Scott was a founding member of the Cork Quality Council (CQC) in 1991. Within the Cork Quality Council and without, Scott has pushed for increased quality control and understanding of cork. In 1994, Scott co-sponsored [with a major winery] a major study regarding the nature of in-vitro development of TCA.
- Later, in the 1990s we worked together with the Cork Quality Council and ETS on a groundbreaking study centered upon using solid phase micro-extraction (SPME) techniques for TCA recognition.

Scott's own search for improved quality standards was also intensified in recent years.

- An all new cork processing facility has been installed offering modern and efficient handling of cork from dedusting to branding to processing to bagging with sulfur dioxide. Corks are stored in a moisture controlled warehouse with microbial growth held in check using ozone.
- On November 30, 1999 Scott's cork facility and laboratory received ISO 9002:1994 certification from the prestigious auditors of TUV Essen. In July 2001 the TUV Essen certified Scott Laboratories, Inc. as a whole as ISO 9001:2000.
- On May 25, 2000, Scott's associated cork facility and laboratory in Portugal [Corval Cortiças] received ISO 9002 certification for its activities.
- In mid-February 2000 Scott Laboratories, Inc. became the first North American firm to complete a survey of its entire cork inventory using SPME technology. This survey included testing samples from each bale by ETS Laboratories [as an independent testing body using Agilent SPME technology]. Since that time Scott has continued SPME screening of all incoming cork shipments. Scott has worked independently and within the CQC to apply increasingly stringent acceptance criteria based upon SPME. Though such efforts have yielded positive reductions in TCA, continued vigilance is required.
- In 2001 Scott purchased an Agilent SPME for use as a pre-screening took in Portugal. This tool has allowed us to move the quality control process closer to the source while continuing SPME testing here.

Scott Laboratories will continue its search to provide the highest quality standards in cork. If you have any questions, please call us. Our technical sales people will be pleased to assist you in your needs.

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FREQUENTLY ASKED QUESTIONS – PACKAGING


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Equipment FAQ

- Corks
- Wirehood

CORKS

Q: What size corks do you offer?
We offer 1 1/2", 1 3/4", 2" and 2 1/4" lengths in various grades and types.

Q: What is the diameter of your cork?
- 24 mm +/- .5 for sterisun and natural corks.
- 23 mm for 1+1 corks.

Q: What is the minimum order quantity?
1,000 cork minimum, sold in 1,000 increments.

Q: What's the best way to store my corks?
Corks should be stored in a cool dry location, not in a bottling room, barrel storage area, or chemical storage area. The temperature should be 55° to 70° F and the humidity 50 to 70%.

Q: How long can I store corks until they are no longer useable?
If corks haven't been opened, and they are stored properly, they are still useable up to 6 months. If corks bags have been opened, they should be returned to a closeable container and sealed tightly. For a minimal charge Scott Laboratories will reprocess, remoisturize and re-SO_2 our corks.

Q: What size cork should I use for an oversized bottle?
We suggest you contact the glass manufacturer for their recommendation.

Q: Do you have a minimum order quantity for oversize corks?
For oversized corks, there is a 50 cork minimum and the order must be placed with another cork order.

Q: Can Corks be printed with custom winery logo?
A printing die is made from customer supplied black and white artwork. Depending on size of cork the artwork should fit into a 34 - 45 mm x 72 mm rectangle.

Q: Where are the price breaks for corks?
5,000, 10,000 and 100,000.

Q: What does Scott Labs do to prevent TCA (cork taint)?
See our Commitment to Quality (PDF) page.

Back to Top

WIREHOODS

Q: What size stock wirehoods are available?
32 and 38 mm all of which are subject to stock on hand.

Q: What is the lead time for custom orders?
Lead time is 12-14 weeks from the time order is placed

Scott Laboratories - Information Center - FAQ - Packaging

Q: How do I determine what size wirehood I need?
Acquire a bottle drawing from the manufacturer and forward it to our attention.

Back to Top

CRDEssay: SPME - Solid Phase Microextraction

Related Concept

- fungi: assessment, sampling and analysis
- fungi: MVOC - microbial volatile organic compound
- VOC: measurement and identification
- measurement: GC-MS
- measurement: SPME

Related References

- Lord, H. L. and Pawliszyn, J., (0). **Recent advances in solid phase microextraction and membrane extraction with a sorbent interface**
- Nilssona, T., Larsenb, T. O., Montanarellic, L. and Madsena, J. Ø., (1996). **Application of head-space solid-phase microextraction for the analysis of volatile metabolites emitted by Penicillium species**
- Pawliszyn, J., (2001). **Solid phase microextraction**

Related Articles

- PCR –polymerase chain reaction

Essay:

SPME - Solid Phase Microextraction

"This is a newly developed technique that is undergoing extensive research, it has been gaining in popularity over the last few years due to: Speed – Equilibration can be reached in only 2-30 minutes – ideal for quick screening. Sensitivity – Parts per trillion detection limits have been attained, with an ion-trap detector. The method which was invented in the early 1990's by Prof. Janusz Pawliszyn from the University of Waterloo in Ontario, Canada, uses a small segment of fused silica fibre coated with an appropriate material such as polydimethylsiloxane, this is then held in a syringe-like device. The extraction of analytes from various matrices is then undertaken and the introduction of these into a chromatographic system for analyses is the final step. No solvents are needed in this process, and analyte extraction and pre-concentration are combined in one single step. SPME fibers is a 1 or sometimes 2cm long fused silica fibre coated with a polymeric phase." -- http://www.chemsoc.org/exemplarchem/entries/2002/Garner/

"Solid-phase microextraction is a sample preparation and sample introduction method in which analytes partition from a sample into a polymer, coated on a fused silica rod of typically 1 cm length by 100 µm diameter. The fibre is fastened into the end of a fine stainless steel tube contained in a syringe-like device, and protected by an outer stainless steel needle. The device's plunger is depressed to expose the fibre to the sample matrix, retracted at the end of the sampling time, and then depressed again to expose the fibre to a desorption interface for analysis, typically by GC or HPLC."-- http://www.science.uwaterloo.ca/chemistry/pawliszyn/Research/MESI/Recentadvances.htm

Chen and Pawliszyn first introduced the technology of coupling standard SPME fibre sampling to HPLC analysis in 1995 6. (J. Chen and J. Pawliszyn, Anal. Chem. 67(15), 2530-2533 (1995)).

SPME is an extraction technology that combines sampling and sample preparation. Since it conception, SPME has been widely used for research applications in pharmaceutical, food, aroma, forensic, environmental and physicochemical properties. Three books on SPME summarize the theory, applications and many practical considerations (1. J. Pawliszyn , Solid Phase Microextraction- Theory and Practice. , Wiley-VCH, New York, USA (1997). 2. J. Pawliszyn (Editor), Applications of Solid Phase Microextraction, The Royal Society of Chemistry, Hertfordshire, UK, 1999. 3. S.A. Scheppers-Wiercinski (Editor), Solid Phase Microextraction: A Practical Guide, Marcel Dekke, New York, USA, 1999) -- Koziel and Novak, 2002, Sampling and sample-preparation strategies based on solid-phase microextraction for analysis of indoor air provided an excellent review

tidbits:

Research publications on SPME at PubMed

Web Links:

PubMed
"service of the National Library of Medicine, includes over 14 million citations for biomedical articles back to the 1950's. .. links to many sites providing full text articles and other related resources"

Solid Phase Microextraction

Brittany Hartzell

A new method in sample preparation should have the following analytical performance characteristics:

- Efficient
- Selective
- Applicable to various compounds and matrices
- Allow for simple automation and field analysis
- Easy to use
- Inexpensive
- Compatible with a wide range of analytical instruments
- Fast
- Use a minimal amount of solvent or be solvent-less
- Few steps

Introduction to SPME

Arthur and Pawliszyn developed this microscale technique in the late 1980's. They introduced it as a solvent-free sample preparation technique that could serve as an alternative to traditional extraction procedures such as liquid-liquid extraction, purge and trap, static headspace, and SPE procedures.

Basic principle: To use a small amount of the extracting phase, usually less than 1μL, while the sample volume can be very large. The extracting phase can be either high molecular weight polymeric liquid (similar to the sp in chromatography) or it can be solid sorbent (typically of high porosity to increase the surface area available for adsorption).

SPE vs. SPME

SPE (solid phase extraction) is a commonly used sorbent extraction technique.

- It is a total extraction technique- *all* of each analyte is transferred to solid extraction phase
- Analytes are extracted together with interfering compounds by passing an aqueous matrix through a plastic cartridge containing dispersed sorbent on a particulate support
- A selective organic solvent is used to remove interferences first, and then another solvent is chosen to wash out the target analytes
- Attractive features: simple, inexpensive, can be used in the field, can be automated, and uses relatively little solvent
- Limitations: low recovery- resulting from interaction between the sample matrix and analytes, some solvent is still necessary, and plugging of the cartridge by solid and oily components

On the other hand, SPME preserves all of the advantages of SPE while eliminating the main disadvantages of low analyte recovery, plugging, and solvent use.

Instrumentation

SPME consists of basically two main steps: 1) Equilibration of the analyte(s) between the fiber coating (extracting phase) and the sample matrix and 2) Desorption of the concentrated analyte(s) into an analytical instrument.

SPME steps in more detail:

1) The fragile fiber is initially withdrawn into the steel syringe needle
2) The sample septum is pierced, the coated fiber is extended into the sample solution for a set time, (typically 2-15 minutes for liquid samples) where the analytes are adsorbed by the fiber until an equilibrium is reached
   - Amount of analyte extracted by coating at equilibrium is determined by the magnitude of the partition coefficient of the analyte between the sample matrix and the coating material
3) The fiber is drawn back into the protective needle and the needle is withdrawn from the sample container
4) The needle is injected into the sample port of an analytical instrument, the fiber is extended, and the analytes are desorbed

SPME Theory

The principle behind SPME is the partitioning of analytes between the sample matrix and extraction medium.

To simplify the theory discussion we can assume the vial containing the sample is completely filled (no headspace is present). If a liquid polymer coating is used, we can use the following equation to relate the amount of analyte adsorbed by the coating at equilibrium to its concentration in the sample:

$$n = \frac{K_{fs} V_f C_o V_s}{K_{fs} V_f + V_s} \qquad \text{Eq. 1}$$

- n: the mass of the analyte absorbed by the coating
- V_f: volume of the coating
- V_s: volume of the sample
- K_{fs}: the distribution constant of the analyte between the coating and the sample matrix
- C_o: the initial concentration of the analyte in the sample

As can be seen from this equation, there exists a linear relationship between the amount of analytes absorbed and their initial concentration in the sample.

Coatings used in SPME typically have strong affinities for organic compounds and therefore, have large K_{fs} values for targeted analytes. This means that SPME is selective and has a very high concentrating effect. However, many times the K_{fs} values are not large enough to exhaustively extract most analytes in the matrix and only through proper calibration can SPME be used to accurately determine concentrations of target analytes. Calibration can be by the external standard method in a relatively clean sample and by standard addition or internal standards in a more complex matrix.

If V_s is very large ($V_s >> K_{fs} V_f$):

$$n = K_{fs} V_f C_o \qquad \text{Eq. 2}$$

This means that when the volume of the sample is very large, the amount of analyte extracted by the fiber coating is not related to the sample volume . This feature, combined with its simple geometry makes SPME ideally suited for field sampling and analysis because the fiber can be exposed to air or dipped directly into a lake or river, without collecting a defined sample volume prior to analysis.

Non-Equilibrium Case:

SPME can still be used for quantitation in this case

$$n = [1 - \exp(-A \frac{2m_1m_2KV_f + 2m_1m_2V_s)}{m_1V_sV_f + 2m_2KV_sV_f}] \frac{KV_fV_s}{KV_f + V_s} C_o$$

A = surface area of polymer coating, m_f = mass transfer coefficient in sample matrix, $m_1 = D_1/\delta_1$, m_2 = mass transfer coefficient in polymer coating, $m_2 = D_2/\delta_2$, D_1, D_2 = diffusion coefficients in sample matrix and polymer coating, δ_1, δ_2 = diffusion layers in sample matrix and polymer coating

This eqn. shows that n is proportional to C_o, if adsorption time and the agitation method is held constant for each sampling.

Kinetics

Perfect Agitation

The liquid or gaseous sample is perfectly agitated- all the analytes present in the sample have access to the fiber coating.

$$t_e = t_{95\%} = \frac{2(b-a)^2}{D_f}$$

Estimate the shortest equilibration time possible by substituting appropriate data for the diffusion coefficient of an analyte in the coating (D_f) and the fiber coating thickness (b-a)

Practical Agitation

Independent of the agitation level, fluid contacting a fiber's surface is always stationary, and as the distance from the fiber surface increases, the fluid movement gradually increases- until it corresponds to bulk flow in the sample. This static layer zone = the boundary layer and it's thickness is determined by the agitation conditions and viscosity of the fluid.

$$t_e = t_{95\%} = 3\ \delta \frac{K_{fs}(b-a)^2}{D_s}$$

(b-a) is the fiber coating's thickness, D_s is the analyte's diffusion coefficient in the sample fluid, K_{fs} is the analyte's distribution constant, δ is the boundary layer thickness.

SPME Sampling

Three basic modes of SPME sampling:

Direct Extraction Mode:

- Coated fiber is inserted into sample and analytes are transported directly from the sample matrix to the extracting phase
- Rapid extraction facilitated by agitation
- For gaseous samples, convection is usually sufficient to facilitate rapid equilibration but for aqueous matrices more efficient agitation needed

Headspace Extraction Mode:

- Analytes extracted from the gas phase that is equilibrated with the sample
- Protects fiber from adverse effects caused by sample matrix
- Allows matrix modifications without affecting the fiber
- Sensitivity is the same as direct extraction, as long as sample and gaseous headspace volumes are the same
- Extraction kinetics are different than in direct extraction mode

Membrane-Protected Mode:

- Fiber is separated from the sample with a selective membrane, lets analytes through while blocking interferences
- Protects fiber from adverse effects caused by sample matrix
- Serves same purpose as headspace mode except that it can still analyze compounds having a low volatility
- Extraction process substantially slower than direct extraction

In-tube SPME (another type of sampling, usually used in the headspace mode)
Extraction phase remains in tubing during extraction- fiber retracted into needle or coating on inner tubing of needle. Therefore, the analyte concentration is measured at a well-defined place in space and time and long-term (integrated) sampling is possible, that accounts for analyte concentration changes with time and place to place variations in field analysis.

SPME: Experimental Variables

Coating Materials

Selectivity can be enhanced by choosing a coated fiber similar in chemical structure to the analyte

- Poly(dimethyl)siloxane- used for alkyl benzenes, PAH's, and volatile halogenated compounds
- Polyacrylate, or mixture of polyacrylate with Carbowax and/or polydivinylbenzene- used for alcohols and small polar compound

Increasing coating thickness, increases V_f and extracts a higher proportion of the analyte

Agitation Methods

As mentioned above, it changes the kinetics and therefore the equilibrium time. Sonication is the best method to reduce t_e.

Salting-out Effect

Addition of an inorganic salt to the aq. sample shifts the partition equilibrium so more analytes are extracted.

Effect of pH

Unless ion exchange coatings are used, SPME can extract only neutral (non-ionic) species. To ensure that at least 99% of acidic compound is in the neutral form, pH should be at least two units lower than pK_a of the analyte and the same goes for a basic compound, the pH should be at least two units higher than the pK_b of the analyte.

Sample Heating

Heating liquid samples gives faster diffusion rates of analytes (to coated surface)- therefore, reducing time needed for equilibrium, but at high temperatures less analyte is extracted. The best method to reduce equilibrium time and still extract sufficient analytes is to use an internally cooled fiber (via an inner capillary of liquid CO_2) while heating the solution.

Derivatization

This experimental variable is necessary for extracting and separating polar compounds. Derivatization can occur by: 1) adding appropriate reagents to matrix, followed by extraction; 2) doping fiber with the reagents, followed by extraction; 3) by extracting then exposing the fiber to derivatizing reagent; 4) by derivatizing within GC inlet.

Interfaces to Analytical Instruments

The GC is used most frequently with SPME because standard GC injections can be applied (without a special interface) as long as a narrow insert exists with an i.d. similar to the o.d of the SPME needle.

SPME/HPLC interface- addresses the need for analysis of nonvolatile and thermally labile analytes. Desorption loop placed in the position where the injection loop normally is. When the injection valve is at the load position, the fiber is introduced into the desorption chamber.

SPME/Optical interface- based on reflectometric interference spectrometry. If any of the extracted analytes strongly absorb the transmitted light, there is a loss in intensity which is detected with a simple optical sensor.

SPME/CE interface- facilitates direct insertion of fiber into inlet so analytes are desorbed directly in capillary, zero dead volume connection is accomplished.

SPME Advantages

- It is an equilibrium technique and is therefore, selective
- Time required for analyte to reach an equilibrium between the coated fiber and sample, relatively short
- Ideal for field sampling: large volume sampling, direct sampling, portable apparatus
- Solvent-less extraction and injection, eliminating solvent disposal
- Smooth liquid coating can be used, eliminating the problem of plugging
- By sampling from headspace, SPME can extract analytes from very complex matrices
- All analytes collected on the solid phase can be injected into GC for further analysis
- Method is fast, inexpensive, and easily automated
- Simple

SPME Disadvantages

- Often only a small fraction of the sample analytes are extracted by the coated fiber
- Quantification in SPME requires calibration
- Carryover resulting from incomplete desorption
- Fiber easily broken
- Limited number of polymeric coatings for SPME- lack of fibers that are sufficiently polar

Applications

Food and Pharmaceuticals:
Advantage in this area is that SPME can extract substances without opening the package. Furthermore, an insignificant amount is extracted and composition of the product does not change.

Environmental:
SPME can meet U.S. EPA method requirements with its low LOD's. These low detection limits reflect that all of the extracted analytes are introduced into the analytical instrument.

Clinical and Forensic:
The major advantage of SPME in this field is its portability: allows for better monitoring of patients during treatment or therapy and better preservation of crime scenes (evidence doesn't need to be taken back to the lab for analysis).